Environmental Laboratory
Licensure Services
(602) 255-3454 (602) 255-1070 FAX
Technical Support Hot-Line 1-800-372-3454
E-Mail: acharyp@azdhs.gov

Information Update

December 22, 1995
Update #21

1. Several of the licensed environmental laboratories have commented on not being able to use palladium as a modifier when analyzing for arsenic or selenium by graphite furnace in wastewaters for NPDES compliance. Palladium nitrate is one of the acceptable modifiers listed in Method 3113B and this method is approved for both arsenic and selenium in wastewaters (40 CFR, part 136.3, Table 1B). The inclusion of this modifier can be found in "Standard Methods, 18th Edition Supplement". This method is also approved for arsenic and selenium in drinking waters (Technical Notes, October 1994).

   Please be certain that your lab is certified for Method 3113B for each parameter in the matrix you wish to analyze (wastewater or drinking water) before using this method.

   Standard Methods, 18th edition supplement, can be ordered from:
   AWWA Bookstore
   6666 West Quincy Avenue
   Denver, Colorado 80235
   Phone: 1-800-926-7337 or (303) 794-7711
   Fax: (303) 794-7310

   PLEASE NOTE THAT EPA 200.9 IS NOT APPROVED FOR THE TESTING OF WASTE WATER FOR NPDES COMPLIANCE TESTING, BUT IT CAN BE USED FOR THE TESTING OF WASTE WATER FOR NON-COMPLIANCE SAMPLES.

2. Following is an outline of the quality control requirements for Method 200.9, Revision 2.2, EMMC version (1994), in drinking water. This outline may not include all the requirements of the method and please be sure to carefully review the whole method before running samples.

   A. Initial demonstration of performance must be completed for all the parameters. This includes the determination of the linear dynamic range and the Method Detection Limits (MDLs). See section 9.2.
   B. . QCS, Quality Control Sample (a secondary source sample) must be within +- 10%. See section 9.2.3.
   C. MDLs should be determined annually, or whenever in the judgement of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be redetermined. See section 9.2.4.
   D. MDLs must be sufficient to detect analytes at the required levels according to compliance

monitoring regulation. See section 9.2.4. See also section 1.2.

E. Analyze one laboratory reagent blank per batch of 20 or fewer samples. See section 9.3.1.
F. Analyze at least one laboratory fortified blank with each batch of samples. Acceptable range is 85-115%. See section 9.3.2.
G. A calibration blank must be run after every calibration, after every 10 samples, and at the end of the sample run. See section 9.3.4. See also comments (aa) from Ted Martin below.
H. The calibration blank should always be less than IDL, Instrument Detection Limit (section 3.5), but greater than the negative signal. See section 9.3.4.
I. A mid level calibration standard IPC, Instrument Performance Check, must be run after every calibration (+- 5% acceptable), and after every tenth sample and at the end of the sample run (+- 10% acceptable). See section 9.3.4.
   Instead of running an IPC standard some laboratories are running a QCS. If a QCS is being run in place of IPC, then the same criteria of +- 5% after the initial calibration must be met. Subsequent analysis of QCS should be within +- 10%. See also comments (bb) from Ted Martin below.
J. All samples must demonstrate a background absorbance of less than 1.0, before the test results obtained can be considered reliable. See section 9.4.1.
K. Analyze a laboratory fortified matrix for a minimum of 10% of the routine samples. Acceptable range is 70-130%. See section 9.4.2 and 9.4.3.
L. The added analyte concentration for lab fortified matrix, must be the same as that used in the laboratory fortified blank. See section 9.4.2.
M. After the warm up period but before the daily calibration, the instrument stability must be demonstrated by analyzing a standard solution with a concentration 20 times the IDL, at a minimum of five times. The resulting relative standard deviation (RSD) of the absorbance signals must be <5%. If the RSD is >5%, determine and correct the cause before calibrating the instrument. See section 11.4.3. See also comments (cc) from Ted Martin below.
N. Determined sample analyte concentrations that are 90% or more of the upper limit of calibration must either be diluted with acidified reagent water and reanalyzed with concern for memory effects or determined by another approved test procedure that is less sensitive. See section 11.4.9. See also comments (dd) from Ted Martin below.
O. Palladium-magnesium modifier must be used. See section 7.7.
P. 95% argon and 5% hydrogen gas mixture must be used. See section 6.1.4.

Following is an outline of comments from a phone conversation with Ted Martin, USEPA/Cincinnati:

AA. The calibration blank is required to verify that the instrumental baseline is not drifting during the analysis.

BB. The control sample may be biased either slightly high or low. If the instrument drifts during the analysis using the control, it may cause the results to go out of control earlier than it should have or it may cause the results to stay in control longer than it should be. He didn't feel that it would be a major problem if the control sample was biased by 1 or 2 percent.

CC. This stability check must be performed.

DD. This section takes into account that the calibration curve extends up to the limit of the linear dynamic range. If the calibration curve is substantially below the limit of this range, then quantification up to the high standard in the calibration curve is allowed.

3. Technical Resources and Training is offering a **FREE** Basic Gas Chromatography workshop. This is being presented by Ms. Jessie Butler, Applications Chemist, Finnigan corporation. Ms. Butler has over 20 years of experience in gas chromatography. This will be held on January 24, 1996 from 9:00 am to 4:00 pm at the Laboratory Licensure office, in the 9th floor conference room, at 3443 N. Central, Phoenix, Arizona 85012. This training will be an overview of gas chromatography. The course will begin with an explanation of the theory of GC covering the principles of separation for packed, Megabore and narrow bore columns. Some historical information will be given on how a solid support is loaded with a stationary phase and how to pack and condition a column or sorbent trap. The importance of the temperature limits of stationary phases and optimization of the parameters for injection with a Megabore versus a narrow bore capillary column will be discussed. Cut away models of inlets will be passed around to demonstrate the various liners available and the importance of guard columns. Special attention will be given to various flow controlling devices available on a gas chromatograph. The presentation will conclude with some tips on keeping your GC "in control" in the ’90s. This class will seat 25 people. Seating is on a first come first serve basis. Please call Cristy Finan at (602) 255-3454 for registration.
4. There will be another **FREE** seminar on "Laboratory Waste Disposal" on February 7, 1996 at the Laboratory Licensure office, 3443 N. Central, Phoenix, Arizona, in the 9th floor conference room, from 9:00 am - 11:30 am. This will be presented by Linda Johnson and Dan Casiraro from the Salt River Project. This presentation will cover an overview of RCRA regulations, laboratory waste streams, waste disposal and laboratory packing. Seating is limited to 35 people. Please call Cristy Finan at (602) 255-3454 for registration.
5. If you have any questions regarding the Updates or if you have any technical questions that need clarification, please call Prabha Acharya, Program Manager, Technical Resources and Training, at the above numbers.